



# Standard Practice for Production of Coal, Coke and Coal Combustion Samples for Interlaboratory Studies<sup>1</sup>

This standard is issued under the fixed designation D6796; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice describes the essential activities that are required to produce samples for interlaboratory studies.

1.2 The suitability of a particular interlaboratory sample developed using this guide will depend on the tests being made.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D121 Terminology of Coal and Coke

D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

D2013 Practice for Preparing Coal Samples for Analysis

D2234/D2234M Practice for Collection of a Gross Sample of Coal

D2492 Test Method for Forms of Sulfur in Coal

D2961 Test Method for Single-Stage Total Moisture Less than 15 % in Coal Reduced to 2.36-mm (No. 8 Sieve) Topsize

D3172 Practice for Proximate Analysis of Coal and Coke

D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke

D3302 Test Method for Total Moisture in Coal

D4596 Practice for Collection of Channel Samples of Coal in a Mine

D5142 Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures

D5263 Test Method for Determining the Relative Degree of Oxidation in Bituminous Coal by Alkali Extraction

E178 Practice for Dealing With Outlying Observations

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E826 Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D121.

## 4. Summary of Practice

4.1 Test samples of adequately defined composition and homogeneity are required for interlaboratory studies. These samples should be typical in all respects to the samples for which the test method is applicable.

4.1.1 The sample should be of sufficient quantity to perform all necessary tests and replicates. Guidelines specifying minimum quantities are given in Practice D2013.

4.1.2 The top size must be consistent for the test being run.

4.2 Differences in results due to the homogeneity or oxidation, or both, of the test sample should be minimal with respect to other errors.

## 5. Significance and Use

5.1 The objective of this practice is to provide guidelines for the preparation of samples for interlaboratory studies to evaluate new test methods or for quality control purposes.

5.2 These samples prepared using these guidelines may be used for the testing of the precision and bias mandatory for ASTM test methods. Such an evaluation is necessary to provide guidance to the user as to the reliability of measurements that can be expected by its use. The statements are developed on the basis of user experience (ordinarily through interlaboratory studies) with the test method.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D05 on Coal and Coke and is the direct responsibility of Subcommittee D05.30 on Quality Assurance.

Current edition approved Feb. 1, 2008. Published March 2008. Originally approved in 2002. Last previous edition approved in 2002 as D6796-02. DOI: 10.1520/D6796-02R08.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

5.3 The availability of appropriate samples is a key requirement for interlaboratory studies.

5.4 The homogeneity of the sample produced for interlaboratory studies must be small enough so that the variance caused by the sample is small compared to the variance of the test being performed.

## 6. Sample Selection and Preparation

6.1 Collect samples using Practice [D2234/D2234M](#), Practice [D4596](#) for coal, or [D346](#) for coke. Coal combustion products should be sampled in a manner to provide a representative sample free from contamination. The sample should be typical of a certain class of samples (for example, Eastern U.S. bituminous coal) or a certain value for a component (such as coals with two percent sulfur), or both, so that appropriate diversity of samples can be identified for interlaboratory studies. See [Note 1](#).

6.2 Grind samples to the proper size required by the test method and properly homogenize and split. Prepare samples using Practice [D2013](#). Some coal combustion products may not require grinding, but tests should be run to determine if further grinding is necessary.

6.3 Both single-laboratory precision and between operator-laboratory precision should be evaluated. Material of adequate homogeneity but of uncertain composition may be used to determine precision of a given test method.

**NOTE 1**—The use of materials of demonstrated adequate homogeneity and known value for the test enables evaluation of the precision and bias of a test method. Materials of known value for the test are required to identify biases. Consensus values obtained as the result of interlaboratory studies are not adequate to permit quantitative evaluation of biases, although they may show that such exist.

6.4 Use six or more materials for each interlaboratory study. See [Notes 1-3](#).

6.5 Each laboratory shall receive its own sample and at least one sample shall be archived by the interlaboratory study coordinator.

6.6 The bulk sample should be available in sufficient quantity for the requirements of the interlaboratory study, to test for homogeneity and to permit further examination by other techniques.

6.7 Sample preparation and distribution should provide enough material and time so that laboratories can perform test runs on new or unfamiliar methods. This is very important because only laboratories that are thoroughly familiar and experienced with the procedure can be considered qualified to participate in the interlaboratory study.

6.8 Sample stability (see [7.6](#)), storage and future accessibility should be considered.

**NOTE 2**—Practice [E691](#) paragraph 10.2.2 states, “An interlaboratory study of a test method should include at least three materials, and for development of broadly applicable precision statements, six or more materials should be included in the study.” Because of the peculiarities of different ranks of coal, more than the minimum six materials are needed to develop precision statements applicable to all coal types. Under no circumstances should the final statement of precision of a test method be based on acceptable test results for each material from fewer than 6 laboratories.

**NOTE 3**—In preparing an interlaboratory sample, the range of values of

the property being measured should be considered, the ranks of coal to which the method will be applied, the difficulty and expense of preparing and distributing samples, the length of time and expense in performing the test and the commercial or legal need for reliable estimates of precision when choosing the number of materials to be used.

## 7. Determining the Suitability of the Sample for the Test Method

7.1 After the sample has been prepared, or if a previously prepared sample is to be used for tests other than for what it was originally prepared, the homogeneity of the sample must be determined.

7.2 Test samples must be sufficiently homogeneous to evaluate the test method. The degree of homogeneity is related to the size of the sample analyzed. Hence the minimum size of a sample meeting a specified homogeneity must be stated, and this must be equal to or smaller than the size of the sample specified in the test method. Also, different tests may require different sample sizes to achieve homogeneity. Tests for homogeneity should be made. An acceptable method for measuring homogeneity is given in [Appendix X1](#).

7.3 The uncertainties in homogeneity, moisture, or sample composition, or a combination thereof, should not exceed one third of the measurement uncertainties they are expected to evaluate. When this specification is met, sample uncertainties make a negligible contribution to the variance of the test results. While replicate samples may be used in practical analysis to average out sample variations, this is not feasible in the case of samples for Interlaboratory Studies; hence, homogeneity should be attained as far as possible.

7.4 Samples not meeting the homogeneity requirements above for a given test should not be used in interlaboratory studies unless they are re-mixed (all samples must be recombined and the entire sample rehomogenized, see [6.2](#)) and retested. In some cases, samples that are inhomogeneous for one test may be homogeneous for other tests.

7.5 Top size of the sample must be consistent with required top size or reduced to the appropriate size.

7.6 Sample stability may be determined. See [Note 4](#).

7.6.1 *Moisture*—For most tests moisture should be determined and compared to other results and calculated to a common basis.

7.6.2 *Oxidation*—Coals (particularly those of low rank) stored for a long period of time may become oxidized. If pyrite becomes oxidized it may affect forms of sulfur determinations (see Test Methods [D2492](#)). Bituminous coals are generally stable for long periods of time but the degree of oxidation of bituminous coals can be determined using Test Method [D5263](#).

## 8. Sample Storage and Preservation

8.1 Samples should be stored in such a way to maintain sample stability for the characteristics being measured. See [Note 4](#).

8.1.1 Changes of values in ultimate analysis should be less than 1 % over a 10 year period.

8.1.2 Moisture should be determined by an appropriate method ([D3172](#), [D3173](#), [D5142](#), [D3302](#), or [D2961](#)) prior to each analysis. It should not be assumed that moisture content will remain constant from day to day especially for low rank coals.

8.1.3 Except for moisture, the largest absolute differences in parameter value changes are likely to be for percent carbon due to oxidation.

8.1.4 For less stable coals or coals with high moisture contents, or both, more aggressive storage or preservation procedures may be employed. See **Note 5**.

8.2 Samples may be conditioned (preoxidized) prior to splitting and bottling. See **Appendix X2**.

8.2.1 Samples may be stored under nitrogen or argon to minimize oxidation. Nitrogen is cheaper but argon is less likely to react with coal components.

8.2.2 Samples may be refrigerated to minimize oxidation. Samples should be maintained at about 4°C. Care must be taken to minimize changes in moisture. It is not recommended that samples be frozen as this might change to pore structure of the coal.

8.2.3 Depending on the characteristics of the coal to be measured, high moisture coals may be stored in water.

**NOTE 4**—Storing samples in darkness or brown bottles can reduce photochemical changes. Air-dried relatively stable-low moisture coals, coke and stable combustion products can be stored in tightly sealed glass or plastic containers at room temperature for long periods of time without significant change of most coal characteristics.

**NOTE 5**—Lower rank coals (subbituminous and lignite) generally have

high moisture content and oxidize rapidly. This oxidation may continue for months but will slow with time. Moisture values in these samples may depend on the time the sample is taken and the relative humidity of the laboratory and may change within seconds after the sample is removed from the bottle and over a longer period of time within the bottle.

## 9. Record Keeping

9.1 Characteristics of the sample should be recorded. At a minimum the rank, percent ash, top size, approximate moisture content, special handling comments, amount of sample available and original purpose for which the standard was collected shall be recorded. Sample location, sample type (run of mine; **D121**, channel sample; **D4596**, etc). Maintain all results with the samples until they are published, at which time only the reference need be maintained.

9.2 Record when the sample was collected, record when the sample was prepared, and record how the sample has been maintained.

9.3 A list of samples currently available with the information listed in **8.1** and **8.2** should be accessible to interested parties who may wish to use them for interlaboratory studies.

## 10. Keywords

10.1 ash; coal; coke; combustion residue; homogeneity; laboratory; moisture; oxidation; quality control

# APPENDIXES

## (Nonmandatory Information)

### X1. TEST FOR HOMOGENEITY

X1.1 Test for homogeneity shall be conducted by a single laboratory in a manner similar to that given in Practice **E826** prior to sample distribution.

X1.1.1 Conduct all tests by the best available procedure under optimum conditions. Preferably a method with a known precision and bias shall be used.

X1.1.2 If the samples have been split into less than 15 laboratory samples, sub-samples from each laboratory sample shall be measured for the test of interest.

X1.1.2.1 Whenever possible the test portions should be taken only after the laboratory samples is rolled in its container at least 5 turns followed by inverting the sample 5 times.

(1) This process should be repeated at least 3 times.

(2) A sample can then be taken from the top.

X1.1.2.2 The steps in **X1.1.2.1** should be repeated for all subsequent test portions even if they are taken from the same container.

X1.1.3 If the number of containers is so large that test portions from each container cannot be measured, a minimum of 8 % but not less than 15 should be tested for homogeneity.

X1.1.4 Label all containers consecutively (that is, 01, 02, 03, ...c). If all sample containers will not be measured (see **X1.1.3**) select the containers to be measured as follows: From a table of random numbers (see **Table X1.1** and the Warning note below ) pick an arbitrary starting place and select any

direction for reading the numbers provided the direction is fixed in advance and is independent of the numbers occurring. Select those containers for testing that match the numbers read from the tables.

**Warning**—Table A1 included herein is for example, only. Use the more complete table in when actually using this test procedure.

X1.1.5 Whenever possible at least 4 separate sub-samples taken from each container and labeled with the container number and sub- sample number (that is, 02-1 for container 02 sub-sample 1). All sub-samples with the same extension (for example, XX-1) shall be determined together as a sample set with appropriate standards and reference materials.

X1.1.6 Examine the data and discard any values that have been determined to be outliers according to Practice **E178**. (See warning note below.) If outliers occur, repeat the complete test, as provisions are not made for missing data in the mathematical treatment.

**Warning**—The use of Practice **E178** dealing with outliers should be done with extreme care to ensure that values are not discarded that may be valid for the analysis.

X1.1.7 When a test is complete, data should be tabulated according to **Table X1.2**. Data may be used in its raw form or normalized to a reference material.

X1.1.8 Compute  $C$ ,  $B$ ,  $c'$ , and  $G$ , (see **Table X1.2**), where:  $C$  = sum of each column;  $B$  = sum of each row;  $c'$  = mean of each column; and  $G$  = sum of  $B_1 \dots B_n$ ;  $b$  = number of replicate measurements (that is, runs); and  $c$  = number of containers.

X1.1.9 Choose a significance level ( $\alpha$ ) for the test (See **Note X1.1**).

NOTE X1.1—A 95 % significance level is recommended for this procedure. See Practice **E178** for more extensive tables containing values at other significance levels.

X1.1.10 From **Table X1.3**, obtain the  $q$  value that corresponds to  $c$  and  $<$ , where:  $<$  = the number of degrees of freedom:

$$< = (b - 1) \times (c - 1) \quad (\text{X1.1})$$

X1.1.11 Compute  $S_t$  = sum of squares due to specimens:

$$S_t = [C_1^2 + C_2^2 + \dots C_c^2]/b - (G^2/cb) \quad (\text{X1.2})$$

X1.1.12 Compute  $S_b$  = sum of squares due to runs:

$$S_b = [B_1^2 + B_2^2 + \dots B_b^2]/c - (G^2/cb) \quad (\text{X1.3})$$

X1.1.13 Compute  $\bar{S}$  = sum of the squares of all the measurements in the tables and subtract  $G^2/cb$ :

$$\bar{S} = (\Gamma_{i=1}^c) (\Gamma_{j=1}^b) Y_{ij}^2 - (G^2/cb) \quad (\text{X1.4})$$

where:

$Y_{ij}$  = individual values in the table.

X1.1.14 Compute the homogeneity factor  $W$ :

$$W = qs/b^{1/2} \quad (\text{X1.5})$$

where:

$$s = [(\bar{S} - S_b - S_c)/(b - 1)(c - 1)]^{1/2}$$

X1.1.15 If the absolute difference between any two mean values (that is  $c'_1 \dots c'_n$ ) exceeds  $W$ , then there is strong evidence, at the 95 % confidence level, that the samples are not homogeneous. If the absolute difference between any two mean values does not exceed  $W$ , then the samples shall be considered to be homogeneous.

**TABLE X1.1 Short Table of Random Numbers<sup>A</sup>**

NOTE—Caution: See warning note in X1.1.4.

46	96	85	77	27	92	86	26	45	21	89	91	71	42	64	64	58	22	75	81	74	91	48	46	18
44	19	15	32	63	55	87	77	33	29	45	00	31	34	84	05	72	90	44	27	78	22	07	62	17
34	39	80	62	24	33	81	67	28	11	34	79	26	35	34	23	09	94	00	80	55	31	63	27	91
74	97	80	30	65	07	71	30	01	84	47	45	89	70	74	13	04	90	51	27	61	34	63	87	44
22	14	61	60	86	38	33	71	13	33	72	08	16	13	50	56	48	51	29	48	30	93	45	66	29
40	03	96	40	03	47	24	60	09	21	21	18	00	05	86	52	85	40	73	73	57	68	36	33	91
52	33	76	44	56	15	47	75	78	73	78	19	87	06	98	47	48	02	62	03	42	05	32	55	02
37	59	20	40	93	17	82	24	19	90	80	87	32	74	59	84	24	49	79	17	23	75	83	42	00
11	02	55	57	48	84	74	36	22	67	19	20	15	92	53	37	13	75	54	89	56	73	23	39	07
10	33	79	26	34	54	71	33	89	74	68	48	23	17	49	18	81	05	52	85	70	05	73	11	17
67	59	28	25	47	89	11	65	65	20	42	23	96	41	64	20	30	89	87	64	37	93	36	96	35
93	50	75	20	09	18	54	34	68	02	54	87	23	05	43	36	98	29	97	93	87	08	30	92	98
24	43	23	72	80	64	34	27	23	46	15	36	10	63	21	59	69	76	02	62	31	62	47	60	34
39	91	63	18	38	27	10	78	88	84	42	32	00	97	92	00	04	94	50	05	75	82	70	80	35
74	62	19	67	54	18	28	92	33	69	98	96	74	35	72	11	68	25	08	95	31	79	11	79	54
91	03	35	60	81	16	61	97	25	14	78	21	22	05	25	47	26	37	80	39	19	06	41	02	00
42	57	66	76	72	91	03	63	48	46	44	01	33	53	62	28	80	59	55	05	02	16	13	17	54
06	36	63	06	15	03	72	38	01	58	25	37	66	48	56	19	56	41	29	28	76	49	74	39	50
92	70	96	70	89	80	87	14	25	49	25	94	62	78	26	15	41	39	48	75	64	69	61	06	38
91	08	88	53	52	13	04	82	23	00	26	36	47	44	04	08	84	80	07	44	76	51	52	41	59
68	85	97	74	47	53	90	05	90	84	87	48	25	01	11	05	45	11	43	15	60	40	31	84	59
59	54	13	09	13	80	42	29	63	03	24	64	12	43	28	10	01	65	62	07	79	83	05	59	61
39	18	32	69	33	46	58	19	34	03	59	28	97	31	02	65	47	47	70	39	74	17	30	22	65
67	43	31	09	12	60	19	57	63	78	11	80	10	97	15	70	04	89	81	78	54	84	87	83	42
61	75	37	19	56	90	75	39	03	56	49	92	72	95	27	52	87	47	12	52	54	62	43	23	13
78	10	91	11	00	63	19	63	74	58	69	03	51	38	60	36	53	56	77	06	69	03	89	91	24
93	23	71	58	09	78	08	03	07	71	79	32	25	19	61	04	40	33	12	06	78	91	97	88	95
37	55	48	82	63	89	92	59	14	72	19	17	22	51	90	20	03	64	96	60	48	01	95	44	84
62	13	11	71	17	23	29	25	13	85	33	35	07	69	25	68	57	92	57	11	84	44	01	33	66
29	89	97	47	03	13	20	86	22	45	59	98	64	53	89	64	94	81	55	87	73	81	58	46	42
16	94	85	82	89	07	17	30	29	89	89	80	98	36	25	36	53	02	49	14	34	03	52	09	20
04	93	10	59	75	12	98	84	60	93	68	16	87	60	11	50	46	56	58	45	88	72	50	46	11
95	71	43	68	97	18	85	17	13	08	00	50	77	50	46	92	45	26	97	21	48	22	23	08	32
86	05	39	14	35	48	68	18	36	57	09	62	40	28	87	08	74	79	91	08	27	12	43	32	03
59	30	60	10	41	31	00	69	63	77	01	89	94	60	19	02	70	88	72	33	38	88	20	60	86
05	45	35	40	54	03	98	96	76	27	77	84	80	08	64	60	44	34	54	24	85	20	85	77	32
71	85	17	74	66	27	85	19	55	56	51	36	48	92	32	44	40	47	10	38	22	52	42	29	96
80	20	32	80	98	00	40	92	57	51	52	83	14	55	31	99	73	23	40	07	64	54	44	99	21
13	50	78	02	73	39	66	82	01	28	67	51	75	66	33	97	47	58	42	44	88	09	28	58	06
67	92	65	41	45	36	77	96	46	21	14	39	56	36	70	15	74	43	62	69	82	30	77	28	77
72	56	73	44	26	04	62	81	15	35	79	26	99	57	28	22	25	94	80	62	95	48	98	23	86
28	86	85	64	94	11	58	78	45	36	34	45	91	38	51	10	68	36	87	81	16	77	30	19	36
69	57	40	80	44	94	60	82	94	93	98	01	48	50	57	69	60	77	69	60	74	22	05	77	17
71	20	03	30	79	25	74	17	78	34	54	45	04	77	42	59	75	78	64	99	37	03	18	03	36
89	98	55	98	22	45	12	49	82	71	57	33	28	69	50	59	15	09	25	79	39	42	84	18	70
58	74	82	81	14	02	01	05	77	94	65	57	70	39	42	48	56	84	31	59	18	70	41	74	60
50	54	73	81	91	07	81	26	25	45	49	61	22	88	41	20	00	15	59	93	51	60	65	65	63
49	33	72	90	10	20	65	28	44	63	95	86	75	78	69	24	41	65	86	10	34	10	32	00	93
11	85	01	43	65	02	85	69	56	88	34	29	64	35	48	15	70	11	77	83	01	34	82	91	04
34	22	46	41	84	74	27	02	57	77	47	93	72	02	95	63	75	74	69	69	61	34	31	92	13

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**TABLE X1.2 Data for Homogeneity Testing**

Run No.	Specimen Number						c <sup>A</sup>	Total
	1	2	3	4	5	...		
1								B <sub>1</sub> =
2								B <sub>2</sub> =
3								B <sub>3</sub> =
4								B <sub>4</sub> =
5...								B <sub>5</sub> =
... b <sup>B</sup>								B <sub>n</sub> =
Total	C <sub>1</sub> =	C <sub>2</sub> =	C <sub>3</sub> =	C <sub>4</sub> =	C <sub>5</sub> =	...	C <sub>n</sub> =	G = (B <sub>1</sub> ... B <sub>n</sub> )
Mean	c <sub>1</sub> '=	c <sub>2</sub> '=	c <sub>3</sub> '=	c <sub>4</sub> '=	c <sub>5</sub> '=	...	c <sub>n</sub> '=	

 A  
B

**TABLE X1.3 Values of  $q$  for Various Combinations for  $c$  and  $v$  at 95 % Significance Level**

$v$ ↓	$c$ →	2	3	4	5	6	7	8	9	10
	1	17.97	26.98	32.82	37.08	40.41	43.12	45.4	47.36	49.07
	2	6.08	8.33	9.8	10.88	11.74	12.44	13.03	13.54	13.99
	3	4.5	5.91	6.82	7.5	8.04	8.48	8.85	9.18	9.46
	4	3.93	5.04	5.76	6.29	6.71	7.05	7.35	7.6	7.83
	5	3.64	4.6	5.22	5.67	6.03	6.33	6.58	6.8	6.99
	6	3.46	4.34	4.9	5.3	5.63	5.9	6.12	6.32	6.49
	7	3.34	4.16	4.68	5.06	5.36	5.61	5.82	6	6.16
	8	3.26	4.04	4.53	4.89	5.17	5.4	5.6	5.77	5.92
	9	3.2	3.95	4.41	4.76	5.02	5.24	5.43	5.59	5.74
	10	3.15	3.88	4.33	4.65	4.91	5.12	5.3	5.46	5.6
	11	3.11	3.82	4.26	4.57	4.82	5.03	5.2	5.35	5.49
	12	3.08	3.77	4.2	4.51	4.75	4.95	5.12	5.27	5.39
	13	3.06	3.73	4.15	4.45	4.69	4.88	5.05	5.19	5.32
	14	3.03	3.7	4.11	4.41	4.64	4.83	4.99	5.13	5.25
	15	3.01	3.67	4.08	4.37	4.59	4.78	4.94	5.08	5.2
	16	3	3.65	4.05	4.33	4.56	4.74	4.9	5.03	5.15
	17	2.98	3.63	4.02	4.3	4.52	4.7	4.86	4.99	5.11
	18	2.97	3.61	4	4.28	4.49	4.67	4.82	4.96	5.07
	19	2.96	3.59	3.98	4.25	4.47	4.65	4.79	4.92	5.04
	20	2.95	3.58	3.96	4.23	4.45	4.62	4.77	4.9	5.01
	24	2.92	3.53	3.9	4.17	4.37	4.54	4.68	4.81	4.92
	30	2.89	3.49	3.85	4.1	4.3	4.46	4.6	4.72	4.82
	40	2.86	3.44	3.79	4.04	4.23	4.39	4.52	4.63	4.73
	60	2.83	3.4	3.74	3.98	4.16	4.31	4.44	4.55	4.65
	120	2.8	3.36	3.68	3.92	4.1	4.24	4.36	4.47	4.56
	$\infty$	2.77	3.31	3.63	3.86	4.03	4.17	4.29	4.39	4.47

## X2. CONDITIONING OF LOW RANK COALS

### X2.1 Statement of Problem:

X2.1.1 Lower rank coals (subbituminous coal and lignite) oxidize faster as more surface area is exposed.

X2.1.1.1 Eight-mesh coal oxidizes faster than as coarser coal, and 60-mesh coal oxidizes faster than 8-mesh coal.

X2.1.1.2 Oxidation may take place even in sealed bottles in a matter of days to months and at a more rapid pace when the samples are exposed to air.

X2.1.1.3 As oxidation progresses, the coal will tend to be more stable.

X2.1.2 For interlaboratory analysis it is not practical that all analyses be done simultaneously or under the same relative humidity.

X2.1.2.1 Laboratories may receive or analyze, or both, samples at different times and under different laboratory conditions.

X2.1.2.2 It is important for interlaboratory studies that differences in analysis be due to analytical performance and not differences in the sample due to coal degradation.

### X2.2 Procedure for Conditioning Low Rank Coals:

X2.2.1 Prepare a large quantity of a consistent quality low rank coal by grinding to the final mesh size for the interlaboratory sample.

X2.2.2 Date the sample, allow it to air dry for 3 to 6 months at a depth of not more than 1 in., and stir frequently to allow for an even oxidation of the material.

X2.2.3 Homogenize and split the entire mass after the drying process is complete (see 6.2).

X2.2.4 Fill, seal and label bottles.

NOTE X2.1—Precision measured with conditioned coals is the precision of the method and this precision may not be attainable for non-conditioned coals as generally tested in industrial applications. Therefore, it is important that precision and bias statements clearly state whether or not conditioned coals were used. In some cases, precision and bias statements can be determined for both conditioned and non-conditioned coals separately in interlaboratory studies.

 **D6796 – 02 (2008)**

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